COMMERCIAL-SCALE DEMONSTRATION OF THE LIQUID PHASE METHANOL (LPMEOHTM) PROCESS

TECHNICAL PROGRESS REPORT NO. 33

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and

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for the Air Products Liquid Phase Conversion Company, L.P.

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Abstract

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was built at a site located at the Eastman chemicals-from-coal complex in Kingsport.

The LPMEOHTM Demonstration Unit operated at 100% availability during this quarter. (Availability is defined as the percentage of time that the LPMEOHTM Demonstration Unit was available to operate, with the exclusion of scheduled outages.) There were two syngas interruptions that were experienced on 18 August 2002 (16.1 hours duration) and 19 August 2002 (12.4 hours duration).

During this quarter, the LPMEOHTM Demonstration Unit was operated at a reactor temperature of 214-215°C and a pressure of 450 psig. The flowrate of the primary syngas feed (Balanced Gas) was controlled at an average value of 551 KSCFH during this time. These conditions have been used since the start of the current catalyst campaign and were selected as a result of the excellent initial activity of the methanol synthesis catalyst following the in-situ activation in June of 2002.

A catalyst deactivation rate of 0.52% per day was calculated for the period 28 June 2002 to 02 August 2002 (35 days). These results are excellent since this deactivation rate includes the period of hyperactivity that is normally encountered with freshly activated methanol synthesis catalysts.

Beginning on 05 August 2002, Balanced Gas composition began to vary greatly for the remainder of the reporting period. This compositional variation resulted in periods of operation with both carbon monoxide (CO)-rich and hydrogen (H₂)-rich syngas. Due to these variations in concentration and their impact on the accuracy of the kinetic model, a catalyst deactivation rate could not be calculated during August and September of 2002. However, it was estimated that the overall catalyst deactivation rate during the three months of operation since the completion of the second in-situ activation was about 0.2% to 0.3% per day. This is a significantly lower deactivation rate than the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day), and may be related to the selection of operating conditions, the performance of the adsorbent in the 29C-40 catalyst guard bed, and the removal of carbon steel components from the reactor during an inspection that was performed in June of 2002. As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte Alternative Fuels Development Unit (AFDU) in 1988/89 was 0.4% per day. (This run was performed on CO-rich syngas derived from natural gas at a reactor temperature of 250°C.)

Due to this low catalyst deactivation rate, the practice of temperature programming was not required during the reporting period (temperature programming involves the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor volumetric productivity).

As noted in Technical Progress Report No. 32, a fresh quantity of adsorbent (activated carbon impregnated with copper oxide) was charged to the 29C-40 catalyst guard bed in June of 2002. This material was chemically reduced using dilute syngas in nitrogen. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or H_2 to copper metal and either carbon dioxide (CO_2) or water (H_2O) in a temperature controlled manner). The catalyst guard bed was placed into service on 27 June 2002.

During the period of 27 June 2002 to 03 July 2002, gas sampling was performed to assess the initial performance of the adsorbent. The analytical techniques involved analyzing the guard bed outlet stream for arsenic (reported as arsine) using standard techniques. The outlet concentration of arsine was determined to be less than the detection limit of the analysis (2 parts per billion by volume (ppbv)). These results provide evidence that the catalyst guard bed was performing well during the first week of operation.

Based upon results over the past year, an operating schedule for the adsorbent was developed. Using this defined timetable, a thermal treatment was completed on 29 August 2002 to provide increased capacity of the adsorbent for arsine removal. The catalyst guard bed was brought back onstream on 30 August 2002. It is planned to replace the used material with a fresh charge in early October of 2002.

The performance of the gas sparger, which was designed by Air Products and first installed into the LPMEOHTM Reactor prior to the restart of the LPMEOHTM Demonstration Unit in March of 1999, was monitored. The device had been inspected and cleaned during the June 2002 outage prior to the second in-situ activation of methanol synthesis catalyst. The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device. The performance of the sparger will continue to be monitored closely for any changes.

During the reporting period, a total of 4,572,193 gallons of methanol was produced at the LPMEOHTM Demonstration Unit. Since startup, about 99.8 million gallons of methanol have been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

A revision to a draft report that provides publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport was sent to DOE for comment. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems.

One hundred percent (100%) of the \$40 million of funds forecast for the Kingsport portion of the LPMEOHTM Process Demonstration Project for the Phase 1 and Phase 2 tasks have

been expended (as invoiced), as of 30 September 2002. Ninety-eight percent (98%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2002.

Table of Contents

Abstract	. 3
Acronyms and Definitions	. 7
Executive Summary	. 9
A. Introduction	
B. Project Description	. 13
C. Process Description	
D. Results and Discussion	. 14
D.1 Off-Site Testing (Product-Use Demonstration)	. 14
D.2 DME Design Verification Testing	
D.3 LPMEOH [™] Process Demonstration Unit - Methanol Operation	
D.4 Planning and Administration	
E. Planned Activities for the Next Quarter	
F. Conclusion	.21
APPENDICES	. 23
APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM	. 23
APPENDIX B - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS	. 24
APPENDIX C - RESULTS OF DEMONSTRATION UNIT OPERATION	. 25
APPENDIX D - MILESTONE SCHEDULE STATUS AND COST	
MANAGEMENT REPORTS	. 29

ACRONYMS AND DEFINITIONS

Air Products - Air Products and Chemicals, Inc.

AFDU - Alternative Fuels Development Unit - The "LaPorte PDU"

AFFTU - Alternative Fuels Field Trailer Unit

Availability - The percentage of time that the LPMEOHTM Demonstration Unit was able to operate, with

the exclusion of scheduled outages

Balanced Gas - A syngas with a composition of hydrogen (H₂), carbon monoxide (CO), and

carbon dioxide (CO₂) in stoichiometric balance for the production of methanol

Btu - British Thermal Unit

Carbon Monoxide Gas - A syngas containing primarily carbon monoxide (CO); also called CO Gas

Catalyst Activity - the rate at which the catalyst promotes the desired chemical reaction to proceed within

the limitations of chemical equilibrium

Catalyst Age $(\eta - eta)$ - the ratio of the rate constant at any point in time to the rate constant for a freshly reduced

catalyst (as determined in the laboratory autoclave)

Catalyst Concentration - Synonym for Slurry Concentration
Catalyst Loading - Synonym for Slurry Concentration

CO Conversion - the percentage of CO consumed across the reactor

Crude Grade Methanol - Underflow from rectifier column (29C-20), defined as 80 wt% minimum purity;

requires further distillation in existing Eastman equipment prior to use

DME - dimethyl ether

DOE - United States Department of Energy

DOE-NETL - The DOE's National Energy Technology Laboratory (Project Team)

DOE-HQ - The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team)
DTP - Demonstration Test Plan - The Operating Plan for Phase 3, Task 2 Operation

DVT - Design Verification Testing
Eastman - Eastman Chemical Company
EIV - Environmental Information Volume
EMP - Environmental Monitoring Plan
EPRI - Electric Power Research Institute

FFV - flexible-fuel vehicle

Fresh Feed - sum of Balanced Gas, H₂ Gas, and CO Gas

Gas Holdup - the percentage of reactor volume up to the Gassed Slurry Height which is gas

Gassed Slurry

Height - height of gassed slurry in the reactor

HAPs - Hazardous Air Pollutants

Hydrogen Gas - A syngas containing an excess of hydrogen (H₂) over the stoichiometric balance for

the production of methanol; also called H₂ Gas

IGCC - Integrated Gasification Combined Cycle, a type of electric power generation plant
 IGCC/OTM - An IGCC plant with a "Once-Thru Methanol" plant (the LPMEOH™ Process) added-on

Inlet Superficial

Velocity - the ratio of the actual cubic feet of gas at the reactor inlet (calculated at the reactor

temperature and pressure) to the reactor cross-sectional area (excluding the area contribution

by the internal heat exchanger); typical units are feet per second

Sparger resistance coefficient (term used in calculation of pressure drop)

KSCFH - Thousand Standard Cubic Feet per Hour

LaPorte PDU - The DOE-owned experimental unit (PDU) located adjacent to Air Products' industrial

gas facility at LaPorte, Texas, where the LPMEOH™ Process was successfully piloted

LPDME - Liquid Phase DME Process, for the production of DME as a mixed coproduct with

methanol

LPMEOH™ - Liquid Phase Methanol (the technology to be demonstrated)

M85 - a fuel blend of 85 volume percent methanol and 15 volume percent unleaded gasoline

MeOH - methanol

Methanol Productivity - the gram-moles of methanol produced per hour per kilogram catalyst (on an oxide basis)

MW - molecular weight, pound per pound mole

ACRONYMS AND DEFINITIONS (cont'd)

NEPA - National Environmental Policy Act

OSHA - Occupational Safety and Health Administration

ρ - density, pounds per cubic foot

Partnership - Air Products Liquid Phase Conversion Company, L.P.

PDU - Process Development Unit
PFD - Process Flow Diagram(s)
ppbv - parts per billion (volume basis)
ppmw - parts per million (weight basis)

Project - Production of Methanol/DME Using the LPMEOH™ Process at an

Integrated Coal Gasification Facility

psi - pounds per square inch

psia
 pounds per square inch (absolute)
 psig
 pounds per square inch (gauge)
 P&ID
 Piping and Instrumentation Diagram(s)

Raw Methanol - sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol

which is produced after stabilization

Reactor Feed - sum of Fresh Feed and Recycle Gas

Reactor O-T-M

Conversion - percentage of energy (on a lower heating value basis) in the Reactor Feed converted to

methanol (Once-Through-Methanol basis)

Reactor Volumetric

Productivity - the quantity of Raw Methanol produced (tons per day) per cubic foot of reactor volume

up to the Gassed Slurry Level

Recycle Gas - the portion of unreacted syngas effluent from the reactor "recycled" as a feed gas Refined Grade Methanol - Distilled methanol, defined as 99.8 wt% minimum purity; used directly in downstream

Eastman processes

SCF - Standard Cubic Feet

SCFH - Standard Cubic Feet per Hour

Slurry Concentration - percentage of weight of slurry (solid plus liquid) which is catalyst (on an oxide basis)

Sl/hr-kg - Standard Liter(s) per Hour per Kilogram of Catalyst

Syngas - Abbreviation for Synthesis Gas

Syngas Utilization - defined as the number of standard cubic feet of Balanced Gas plus CO Gas to the

LPMEOHTM Demonstration Unit required to produce one pound of Raw Methanol

Synthesis Gas - a gas containing primarily hydrogen (H₂) and carbon monoxide (CO), or mixtures of

 \boldsymbol{H}_2 and CO; intended for "synthesis" in a reactor to form methanol and/or other

hydrocarbons (synthesis gas may also contain CO₂, water, and other gases)

Temperature

Programming - the increase of reactor temperature as necessary to control the reactor purge flowrate and

maintain reactor volumetric productivity

Tie-in(s) - the interconnection(s) between the LPMEOH™ Process Demonstration

Unit and the Eastman Facility

TPD - Ton(s) per Day

V - volumetric flowrate, thousand standard cubic feet per hour

VOC - volatile organic compound

vol% - volume percent

WBS - Work Breakdown Structure

wt - weight

Executive Summary

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was designed, constructed, and is in operation at a site located at the Eastman chemicals-from-coal complex in Kingsport.

On 04 October 1994, Air Products and Eastman signed the agreements that would form the Partnership, secure the demonstration site, and provide the financial commitment and overall project management for the project. These partnership agreements became effective on 15 March 1995, when DOE authorized the commencement of Budget Period No. 2 (Modification No. A008 to the Cooperative Agreement). The Partnership has subcontracted with Air Products to provide the overall management of the project, and to act as the primary interface with DOE. As subcontractor to the Partnership, Air Products provided the engineering design, procurement, construction, and commissioning of the LPMEOHTM Process Demonstration Unit, and is providing the technical and engineering supervision needed to conduct the operational testing program required as part of the project. As subcontractor to Air Products, Eastman is responsible for operation of the LPMEOHTM Process Demonstration Unit, and for the interconnection and supply of syngas, utilities, product storage, and other needed services.

The project involves the operation of an 80,000 gallons per day (260 tons per day (TPD)) methanol unit utilizing coal-derived syngas from Eastman's integrated coal gasification facility. The new equipment consists of syngas feed preparation and compression facilities, the liquid phase reactor and auxiliaries, product distillation facilities, and utilities.

The technology to be demonstrated is the product of a cooperative development effort by Air Products and DOE in a program that started in 1981. Developed to enhance electric power generation using integrated gasification combined cycle (IGCC) technology, the LPMEOH™ Process is ideally suited for directly processing gases produced by modern day coal gasifiers. Originally tested at the Alternative Fuels Development Unit (AFDU), a small, DOE-owned experimental unit in LaPorte, Texas, the technology provides several improvements essential for the economic coproduction of methanol and electricity directly from gasified coal. This liquid phase process suspends fine catalyst particles in an inert liquid, forming a slurry. The slurry dissipates the heat of the chemical reaction away from the catalyst surface, protecting the catalyst and allowing the methanol synthesis reaction to proceed at higher rates.

At the Eastman chemicals-from-coal complex, the technology is integrated with existing coal gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable

on/off operation, and its ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site, product-use test program was conducted to demonstrate the suitability of the methanol product as a transportation fuel and as a fuel for stationary applications for small modular electric power generators for distributed power.

The operating test phase and the completed off-site product-use test program have been developed to demonstrate the commercial viability of the LPMEOH™ Process and allow utilities to evaluate the application of this technology in the coproduction of methanol with electricity. A typical commercial-scale IGCC coproduction facility, for example, could be expected to generate 200 to 350 MW of electricity, and to also manufacture 45,000 to 300,000 gallons per day of methanol (150 to 1,000 TPD). A successful demonstration at Kingsport will show the ability of a local resource (coal) to be converted in a reliable (storable) and environmentally preferable way to provide the clean energy needs of local communities for electric power and transportation.

This project has also completed design verification testing (DVT), including laboratory- and pilot-scale research and market verification studies, to evaluate whether to include a demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol. DME has several commercial uses. In a storable blend with methanol, the mixture can be used as a peaking fuel in gasification-based electric power generating facilities, or as a diesel engine fuel. Blends of methanol and DME can be used as chemical feedstocks for synthesizing chemicals, including new oxygenated fuel additives.

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March of 1995 and formally approved it on 01 June 1995 (Modification No. M009). After approval, the project initiated Phase 1 - Design activities. Phase 2 - Construction - activities were initiated in October of 1995. The project required review under the National Environmental Policy Act (NEPA) to move to the construction phase. DOE prepared an Environmental Assessment (DOE/EA-1029), and subsequently a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. The Cooperative Agreement was modified (Modification No. A011) on 08 October 1996, authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full \$213,700,000 of authorized funding, with 56.7% participant cost share and 43.3% DOE cost share.

The LPMEOHTM Demonstration Unit operated at 100% availability during this quarter. (Availability is defined as the percentage of time that the LPMEOHTM Demonstration Unit was available to operate, with the exclusion of scheduled outages.) There were two syngas interruptions that were experienced on 18 August 2002 (16.1 hours duration) and 19 August 2002 (12.4 hours duration).

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Due to this low catalyst deactivation rate, the practice of temperature programming was not required during the reporting period (temperature programming involves the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor volumetric productivity).

Samples of spent and recently activated fresh catalyst were collected during the quarter to determine changes in levels of trace contaminants (such as iron, nickel, sulfur, and arsenic) that are known poisons to methanol synthesis catalysts. These samples will be analyzed and reported when available.

As noted in Technical Progress Report No. 32, a fresh quantity of adsorbent (activated carbon impregnated with copper oxide) was charged to the 29C-40 catalyst guard bed in June of 2002. This material was chemically reduced using dilute syngas in nitrogen. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or H_2 to copper metal and either carbon dioxide (CO_2) or water (H_2O) in a temperature controlled manner). The catalyst guard bed was placed into service on 27 June 2002.

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Based upon results over the past year, an operating schedule for the adsorbent was developed. Fresh adsorbent can be used for about two months before breakthrough of arsine can be expected. A thermal treatment can then be performed on the adsorbent to provide increased capacity for arsine removal, and the material can be used for another month before the adsorbent needs to be replaced. Based upon this timetable, a thermal treatment was completed on 29 August 2002. The catalyst guard bed was brought back onstream on 30 August 2002. It is planned to replace the used material with a fresh charge in early October of 2002.

The performance of the gas sparger, which was designed by Air Products and first installed into the LPMEOHTM Reactor prior to the restart of the LPMEOHTM Demonstration Unit in March of 1999, was monitored. The device had been inspected and cleaned during the June 2002 outage prior to the second in-situ activation of methanol synthesis catalyst. The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device. The performance of the sparger will continue to be monitored closely for any changes.

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A. Introduction

The Liquid Phase Methanol (LPMEOH™) demonstration project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L. P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol was designed, constructed, and is operating at a site located at the Eastman chemicals-from-coal complex in Kingsport. The Partnership will own and operate the facility for the demonstration period.

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to "demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility." The project has been demonstrating the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project has also evaluated the demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol.

The LPMEOH[™] Process is the product of a cooperative development effort by Air Products and the DOE in a program that started in 1981. It was successfully piloted at a 10-TPD rate in the DOE-owned experimental unit at Air Products' LaPorte, Texas, site. This demonstration project is the culmination of that extensive cooperative development effort.

B. Project Description

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 8,600 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas or H₂ Gas, carbon monoxide gas or CO Gas, and the primary syngas feed known as Balanced Gas) are diverted from existing operations to the LPMEOH™ Demonstration Unit, thus providing the range of coal-derived syngas ratios (hydrogen to carbon monoxide) needed to meet the technical objectives of the demonstration project.

For descriptive purposes and for design and construction scheduling, the project has been divided into four major process areas with their associated equipment:

- Reaction Area Syngas preparation and methanol synthesis reaction equipment.
- Purification Area Product separation and purification equipment.
- Catalyst Preparation Area Catalyst and slurry preparation and disposal equipment.
- Storage/Utility Area Methanol product, slurry, and oil storage equipment.

The physical appearance of this facility closely resembles the adjacent Eastman process plants, including process equipment in steel structures.

• Reaction Area

The reaction area includes feed gas compressors, catalyst guard beds, the reactor, a steam drum, separators, heat exchangers, and pumps. The equipment is supported by a matrix of structural steel. The most salient feature is the reactor, since with supports, it is approximately 84-feet tall.

• Purification Area

The purification area features two distillation columns with supports; one is approximately 82-feet tall, and the other 97-feet tall. These vessels resemble the columns of the surrounding process areas. In addition to the columns, this area includes the associated reboilers, condensers, air coolers, separators, and pumps.

• Catalyst Preparation Area

The catalyst preparation area consists of a building with a roof and partial walls, in which the catalyst preparation vessels, slurry handling equipment, and spent slurry disposal equipment are housed. In addition, a hot oil utility system is included in the area.

• Storage/Utility Area

The storage/utility area includes two diked lot-tanks for methanol, two tanks for oil storage, a slurry holdup tank, a trailer loading/unloading area, and an underground oil/water separator. A vent stack for safety relief devices is located in this area.

C. Process Description

The LPMEOH™ Demonstration Unit is integrated with Eastman's coal gasification facility. A simplified process flow diagram is included in Appendix A. Syngas is introduced into the slurry reactor, which contains a slurry of liquid mineral oil with suspended solid particles of catalyst. The syngas dissolves through the mineral oil, contacts the catalyst, and reacts to form methanol. The heat of reaction is absorbed by the slurry and is removed from the slurry by steam coils. The methanol vapor leaves the reactor, is condensed to a liquid, sent to the distillation columns for removal of higher alcohols, water, and other impurities, and is then stored in the day tanks for sampling before being sent to Eastman's methanol storage. Most of the unreacted syngas is recycled back to the reactor with the syngas recycle compressor, improving cycle efficiency. The methanol will be used for downstream feedstocks and has been used in off-site, product-use testing to determine its suitability as a transportation fuel and as a fuel for stationary applications in the power industry.

D. Results and Discussion

The project status is reported by task, covering those areas in which activity took place during the reporting period. Major accomplishments during this period are as follows:

D.1 Off-Site Testing (Product-Use Demonstration)

The LPMEOHTM Demonstration Project has completed the testing of stabilized methanol from both the LaPorte AFDU and the Kingsport LPMEOHTM Demonstration Unit in various off-site mobile and stationary applications. The product-use test program was developed to enhance the early commercial acceptance of central clean coal technology processing

facilities, coproducing electricity and methanol to meet the needs of the local community. One of the advantages of the LPMEOHTM Process for coproduction from coal-derived syngas is that the as-produced, stabilized (degassed) methanol product is of unusually high quality (e.g. less than 1 wt% water) which may be suitable for the premium fuel applications. When compared to conventional methanol synthesis processes, cost savings (10 to 15%) of several cents per gallon of methanol can be achieved in coproduction facilities, if the suitability of the stabilized product can be demonstrated.

Product-use tests commenced during the first year of demonstration operations. An inventory of approximately 12,000 gallons of stabilized methanol was produced at LPMEOHTM Demonstration Unit in February 1998 to supply the needs of the product-use test program; due to the pre-1998 timing for certain tests, methanol was shipped from the inventory produced and held at the LaPorte AFDU. The stabilized methanol from the February 1998 production campaign has been stored in an offsite facility; during the reporting period, the unused stabilized methanol was returned to Eastman for further distillation prior to use within the chemicals-from-coal complex.

A Topical Report entitled "Off-Site Testing of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process" has been issued (February 2002). This report provides the results from the seven test sites.

D.2 DME Design Verification Testing

The LPMEOHTM Demonstration Project has completed Design Verification Testing (DVT) to coproduce dimethyl ether (DME) with methanol via the Liquid Phase Dimethyl Ether (LPDME) Process. DVT was required to provide additional data for engineering design and evaluation of the potential for demonstration at the LPMEOHTM Demonstration Unit. The essential steps required for decision-making were: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks.

Execution of the LPDME DVT at the LaPorte AFDU was completed during October and November of 1999, and preliminary results from the operation were presented in Technical Progress Report No. 22. Results from a cost estimate for a commercial-scale LPDME plant were presented in Technical Progress Report No. 23. After discussing the results from the LPDME DVT activities and the ongoing performance results from Kingsport, the project participants agreed that the available resources should be directed toward improving the catalyst performance for the LPMEOHTM Process during the remaining time within the operating program; any improvement in the catalyst performance for the methanol synthesis catalyst will also yield benefits for the LPDME catalyst system.

A Topical Report, which presents the results of the DVT at the LaPorte AFDU, has been issued (March 2001).

A Topical Report, which provides the status of the current market for DME and an outlook on potential market developments through 2006, has been issued (April 2002).

D.3 LPMEOH™ Process Demonstration Unit - Methanol Operation

Table D.3-1 contains the summary table of performance data for the LPMEOHTM Demonstration Unit during the reporting period. These data represent daily averages, typically from a 24-hour material balance period, and those days with less than 12 hours of stable operation are omitted. Appendix B contains samples of the detailed material balance reports, which are representative of the operation of the LPMEOHTM Demonstration Unit during the reporting period.

During the reporting period, a total of 4,572,193 gallons of methanol was produced at the LPMEOHTM Demonstration Unit. Eastman accepted this entire methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents or injuries were reported during this quarter.

The LPMEOHTM Demonstration Unit operated at 100% availability during this quarter. Appendix C, Table 1 contains the summary of outages for the LPMEOHTM Demonstration Unit during this quarter. There were two syngas interruptions that were experienced on 18 August 2002 (16.1 hours duration) and 19 August 2002 (12.4 hours duration).

<u>Catalyst Life (eta) – July - September 2002</u>

The "age" of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable eta (η) , which is defined as the ratio of the rate constant at any time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix C, Figure 1 plots $\log \eta$ versus days onstream for the fourth catalyst campaign (which began in June 2002 following the second in-situ activation of methanol synthesis catalyst in the LPMEOHTM Reactor). Since catalyst activity typically follows a pattern of exponential decay, the plot of $\log \eta$ is fit to a series of straight lines, with step-changes whenever reactor temperature is changed.

During this quarter, the LPMEOHTM Demonstration Unit was operated at a reactor temperature of 214-215°C and a pressure of 450 psig. The flowrate of Balanced Gas was controlled at an average value of 551 KSCFH during this time. These conditions have been used since the start of the current catalyst campaign and were selected as a result of the excellent initial activity of the methanol synthesis catalyst following the in-situ activation in June of 2002.

A catalyst deactivation rate of 0.52% per day was calculated for the period 28 June 2002 to 02 August 2002 (35 days). These results are excellent since this deactivation rate includes the period of hyperactivity that is normally encountered with freshly activated methanol synthesis catalysts.

Beginning on 05 August 2002, Balanced Gas composition began to vary greatly for the remainder of the reporting period. This compositional variation resulted in periods of operation with both CO-rich and H₂-rich syngas. Due to these variations in concentration and their impact on the accuracy of the kinetic model, a catalyst deactivation rate could not be calculated during August and September of 2002. However, it was estimated that the

Table D.3-1. Data Summary for LPMEOHTM Demonstration Unit

																		Reactor		Raw			U		
		Days				Fresh	Recycle	Reactor	Purge	Inlet Sup.	Space	Slurry	Gas	Gassed	Catalyst	Catalyst	CO	O-T-M	Syngas	MeOH	Catalyst	Reactor	Overall	Sparger	Sparger
		On	Gas	Temp	Pres.	Feed	Gas	Feed	Gas	Velocity	Velocity	Conc.	Holdup	Slurry	Inventory	Age	Conv.	Conv.	Util.	Prod.	MeOH Prod.	Vol. Prod.	(Btu	dP	Resist.
Case	Date	Stream	Туре	(Deg C)	(psig)	(KSCFH)	(KSCFH)	(H2:CO)	(KSCFH)	(ft/sec)	(l/hr-kg)	(wt% ox)	(vol%)	Hgt (ft)	(lb)	(eta)	(%)	(%)	(SCF/lb)	(TPD)	(gmol/hr-kg)	(TPD/ft3)	hr ft2 F)	(psi)	("K")
2000-8	1-Jul-02	4	Balanced	214	450	558	1,366	3.82	32.6	0.65	2764	37.8	23.2	50.0	40,040	1.181	2.1	50.3	27.1	39.7	168.70	11.000	0.08	126.00	5.93
2000-8	2-Jul-02 3-Jul-02	5 6	Balanced	214	450	550	1,353	3.91	34.0	0.65	2747	38.5	23.1	48.5	40,040	1.141	3.1	50.5	26.8	39.6	166.70	10.870	0.082	125.00	5.78
2000-8	3-Jul-02 4-Jul-02	7	Balanced	215	450	553	1,353	3.86	35.3	0.65	2755	38.7	22.7	48.0	40,040	1.107	4.1	49.8	26.7	39.7	167.00	10.890	0.083	119.00	5.77
2000-8 2000-8	5-Jul-02	8	Balanced Balanced	214 215	450 450	550 550	1,353 1,337	3.90 3.93	34.4 34.2	0.65	2739 2735	38.4 38.9	23.1 24.0	49.0 48.5	40,040 40,040	1.093 1.115	5.1 6.1	50.2 50.9	26.7 26.8	39.9 39.7	165.60 166.20	10.800 10.830	0.081	113.00 119.00	5.72 5.81
2000-8	6-Jul-02	9	Balanced	215	450	550	1,354	3.85	32.7	0.65	2751	38.2	22.7	49.0	40,040	1.113	7.1	50.9	26.8	39.4	167.30	10.830	0.082	113.00	5.81
2000-8	7-Jul-02	10	Balanced	213	450	550	1,368	3.70	34.2	0.66	2765	38.5	22.7	48.0	40,040	1.102	8.1	48.0	26.5	39.4	166.50	10.910	0.081	118.00	5.92
2000-8	8-Jul-02	11	Balanced	214	450	523	1,389	5.01	27.8	0.65	2763	38.7	21.9	47.5	40,040	1.067	9.1	47.8	26.0	39.0	161.00	10.490	0.083	117.00	6.02
2000-8	9-Jul-02	12	Balanced	214	450	525	1,391	3.69	28.4	0.66	2766	39.4	22.2	46.5	40,040	1.043	10.1	47.0	25.9	39.2	160.80	10.480	0.081	117.00	6.08
2000-8	10-Jul-02	13	Balanced	215	450	522	1,402	3.53	26.6	0.66	2782	39.7	23.1	46.5	40,040	1.023	11.1	45.5	25.9	39.1	160.30	10.450	0.082	115.00	6.28
2000-8	11-Jul-02	14	Balanced	215	450	525	1,434	3.48	26.8	0.67	2828	39.1	22.2	47.0	40,040	1.015	12.1	44.5	25.6	39.1	161.30	10.520	0.082	115.00	6.41
2000-8	12-Jul-02	15	Balanced	215	450	523	1,431	3.40	25.1	0.67	2822	38.8	22.9	48.0	40,040	1.021	13.1	44.2	25.9	38.8	161.60	10.520	0.08	114.00	6.52
2000-8	13-Jul-02	16	Balanced	215	450	522	1,434	3.22	23.0	0.67	2831	39.6	24.5	47.5	40,040	1.020	14.1	42.6	26.0	38.4	163.20	10.620	0.082	115.00	6.60
2000-8	14-Jul-02	17	Balanced	215	450	522	1,417	3.27	22.1	0.67	2814	38.6	23.8	49.0	40,040	1.045	15.1	43.4	26.1	38.6	162.20	10.560	0.079	117.00	6.58
2000-8	15-Jul-02	18	Balanced	215	450	523	1,406	3.46	25.4	0.66	2789	39.1	23.1	47.5	40,040	1.057	16.1	44.8	25.8	38.9	161.40	10.510	0.081	125.00	6.26
2000-8	16-Jul-02	19	Balanced	215	750	523	1,418	3.34	23.5	0.66	2806	37.8	23.1	50.0	40,040	1.073	17.1	44.1	26.1	38.6	162.60	10.580	0.077	124.00	6.52
2000-8	17-Jul-02	20	Balanced	215	450	523	1,407	3.39	25.4	0.66	2791	37.7	21.9	49.5	40,040	1.039	18.1	44.0	25.8	38.9	161.10	10.490	0.078	118.00	6.36
2000-8	18-Jul-02	21	Balanced	215	450	522	1,420	3.15	24.9	0.66	2805	38.8	23.1	48.0	40,040	1.027	19.1	41.9	25.9	38.8	161.70	10.530	0.08	122.00	6.65
2000-8	19-Jul-02	22	Balanced	215	450	522	1,404	3.33	23.9	0.66	2793	39.0	25.2	49.0	40,040	1.067	20.1	44.1	26.2	38.8	161.70	10.530	0.079	130.00	6.48
2000-8	20-Jul-02	23	Balanced	215	450	523	1,410	3.31	23.6	0.67	2793	38.5	25.0	50.0	40,040	1.020	21.1	43.9	26.2	38.7	161.90	10.540	0.077	112.00	6.53
2000-8	21-Jul-02	24	Balanced	215	450	523	1,397	3.46	24.8	0.65	2769	37.5	23.0	50.5	40,040	1.063	22.1	45.1	26.0	38.9	161.30	10.500	0.076	123.00	6.26
2000-8	22-Jul-02	25	Balanced	215	450	523	1,396	3.48	24.8	0.66	2771	37.9	23.4	50.0	40,040	1.055	23.1	45.1	25.9	38.9	161.10	10.490	0.077	124.00	6.24
2000-8	23-Jul-02	26	Balanced	215	450	520	1,395	3.46	24.4	0.66	2773	38.5	24.6	50.5	40,040	1.044	24.1	44.8	25.9	38.9	160.60	10.460	0.076	125.00	6.28
2000-8	24-Jul-02	27	Balanced	215	450	523	1,399	3.34	26.0	0.66	2782	38.2	23.5	49.5	40,040	1.027	25.1	43.6	25.9	38.9	161.30	10.510	0.078	124.00	6.34
2000-8	25-Jul-02	28	Balanced	215	450	520	1,410	3.19	25.9	0.66	2790	38.6	23.1	48.5	40,040	1.005	26.1	41.8	25.7	38.9	160.50	10.450	0.079	122.00	6.42
2000-8	26-Jul-02	29	Balanced	215	450	520	1,408	3.16	26.6	0.66	2785	38.8	22.1	47.5	40,040	0.996	27.1	41.4	25.6	38.9	160.50	10.450	0.081	121.00	6.36
2000-8	27-Jul-02	30	Balanced	214	450	520	1,407	3.14	27.9	0.66	2774	39.0	21.9	47.0	40,040	0.980	28.1	40.9	25.4	39.1	159.60	10.390	0.081	119.00	6.32
2000-8	28-Jul-02 30-Jul-02	31 33	Balanced	215	450	518	1,401	3.21	26.9	0.65	2767	38.7	21.8	47.5	40,040	0.988	29.1	41.7	25.5	39.0	159.40	10.380	0.08	120.00	6.25
2000-8 2000-8	31-Jul-02	34	Balanced	215	450	520	1,401	3.23	26.5	0.66	2772	38.2	22.8	49.0	40,040	0.999	31.1	42.0	25.6	39.1	159.50	10.390	0.078	121.00	6.29
	1-Aug-02	35	Balanced	215	450	520	1,401	3.27	29.6	0.65	2766	37.7	21.2	49.0	40,040	0.984	32.1	42.2	21.2	39.3	158.80	10.340	0.077	117.00	6.24
2000-8 2000-8	2-Aug-02	36	Balanced Balanced	215 215	450 450	518 518	1,408 1,410	3.15 3.07	29.2 28.5	0.65	2771 2780	38.9 38.2	24.0 23.6	48.5 49.5	40,040 40,040	0.986 0.976	33.1 34.1	41.4 40.5	25.6 25.6	39.1 39.1	159.00 158.90	10.360 10.350	0.078	124.00 122.00	6.43 6.55
2000-8	3-Aug-02	37	Balanced	215	450	518	1,410	3.34	27.8	0.65	2742	37.3	23.6	50.5	40,040	1.011	35.1	40.5	25.8	39.1	158.90	10.350	0.076	121.00	6.06
2000-8	4-Aug-02	38	Balanced	215	450	518	1,412	2.94	25.3	0.66	2788	37.7	22.9	50.0	40,040	1.002	36.1	39.8	25.8	38.5	161.60	10.430	0.077	122.00	6.56
2000-8	9-Aug-02	43	Balanced	215	450	528	1,323	5.37	32.4	0.63	2664	37.9	22.7	49.5	40,040	1.037	37.1	48.8	26.3	39.4	160.80	10.480	0.077	124.00	5.35
2000-8	13-Aug-02	47	Balanced	214	450	527	1,224	4.62	59.3	0.59	2518	38.1	20.1	47.5	40,040	1.018	38.1	54.9	25.7	41.5	152.40	9.940	0.076	123.00	4.42
2000-8	14-Aug-02	48	Balanced	214	450	532	1,194	4.77	69.3	0.59	2484	36.8	18.5	49.0	40,040	1.015	39.1	56.0	25.6	42.5	150.30	9.810	0.073	117.00	4.27
2000-8	15-Aug-02	49	Balanced	215	450	528	1,215	4.62	58.2	0.60	2520	37.8	20.7	48.5	40,040	1.014	40.1	54.9	25.7	41.6	152.20	9.920	0.073	119.00	4.46
2000-8	16-Aug-02	50	Balanced	215	450	530	1,235	4.52	55.6	0.60	2546	38.0	21.4	48.5	40,040	1.019	41.1	54.2	25.8	41.3	154.00	10.040	0.076	123.00	4.58
2000-8	17-Aug-02	51	Balanced	215	450	529	1,271	4.23	49.3	0.61	2595	37.7	21.3	49.0	40,040	1.007	42.1	51.5	25.8	40.8	155.70	10.150	0.076	123.00	4.90
2000-8	28-Aug-02	62	Balanced	215	450	569	1,339	3.41	47.0	0.65	2766	38.3	21.5	48.0	40,040	0.999	43.1	44.5	26.4	40.5	168.80	11.000	0.084	118.00	5.04
2000-8	6-Sep-02	71	Balanced	215	450	583	1,279	3.99	72.0	0.64	2695	38.3	21.4	48.0	40,040	0.992	44.1	49.2	25.9	42.3	165.30	10.790	0.082	119.00	4.44
2000-8	7-Sep-02	72	Balanced	215	450	583	1,306	3.68	66.6	0.65	2732	38.3	21.5	48.0	40,040	0.976	45.1	46.2	25.9	42.0	166.40	10.860	0.083	119.00	4.69
2000-8	8-Sep-02	73	Balanced	215	450	578	1,287	3.84	72.2	0.64	2702	38.8	21.3	47.0	40,040	0.960	46.1	47.2	25.6	42.4	163.70	10.690	0.083	119.00	4.51
2000-8	9-Sep-02	74	Balanced	215	450	588	1,309	3.66	68.4	0.65	2736	38.4	21.9	48.0	40,040	1.005	47.1	46.8	26.3	41.7	169.10	11.040	0.084	123.00	4.73
2000-8	10-Sep-02	75	Balanced	215	450	588	1,279	3.61	67.0	0.64	2726	38.8	22.3	47.5	40,040	1.011	48.1	46.7	26.6	41.7	169.20	11.040	0.085	125.00	4.72
2000-8	11-Sep-02	76	Balanced	214	450	586	1,223	4.05	80.6	0.62	2638	38.8	21.5	47.0	40,040	0.992	49.1	50.1	26.1	42.8	164.10	10.720	0.083	121.00	4.16
2000-8	12-Sep-02	77	Balanced	214	450	594	1,256	3.95	73.2	0.63	2666	38.9	21.7	47.0	40,040	1.027	50.1	50.2	26.8	41.9	169.90	11.080	0.086	125.00	4.28
2000-8	14-Sep-02	79	Balanced	215	450	593	1,189	4.19	77.0	0.62	2610	37.8	22.3	49.5	40,040	1.044	52.1	52.8	26.8	42.6	167.30	10.920	0.081	123.00	4.07
2000-8	15-Sep-02	80	Balanced	215	450	593	1,223	3.99	71.2	0.62	2643	38.0	23.0	49.5	40,040	1.031	53.1	50.9	26.9	42.1	169.00	11.030	0.081	123.00	4.25
2000-8	16-Sep-02	81	Balanced	215	450	593	1,206	4.26	76.7	0.62	2419	38.1	22.4	49.0	40,040	1.036	54.1	51.8	26.8	42.4	167.80	10.950	0.082	123.00	4.11
2000-8	17-Sep-02	82	Balanced	215	450	598	1,232	3.92	71.9	0.63	2656	38.1	22.4	49.0	40,040	1.020	55.1	50.2	26.8	42.4	169.40	11.050	0.082	121.00	4.28

Table D.3-1. Data Summary for LPMEOH $^{\text{TM}}$ Demonstration Unit (continued)

																		Reactor		Raw			U		
		Days				Fresh	Recycle	Reactor	Purge	Inlet Sup.	Space	Slurry	Gas	Gassed	Catalyst	Catalyst	CO	O-T-M	Syngas	MeOH	Catalyst	Reactor	Overall	Sparger	Sparger
		On	Gas	Temp	Pres.	Feed	Gas	Feed	Gas	Velocity	Velocity	Conc.	Holdup	Slurry	Inventory	Age	Conv.	Conv.	Util.	Prod.	MeOH Prod.	Vol. Prod.	(Btu	dP	Resist.
Case	Date	Stream	Type	(Deg C)	(psig)	(KSCFH)	(KSCFH)	(H2:CO)	(KSCFH)	(ft/sec)	(l/hr-kg)	(wt% ox)	(vol%)	Hgt (ft)	(lb)	(eta)	(%)	(%)	(SCF/lb)	(TPD)	(gmol/hr-kg)	(TPD/ft3)	hr ft2 F)	(psi)	("K")
2000-8	18-Sep-02	83	Balanced	215	450	591	1,295	3.33	58.3	0.65	2749	38.5	23.8	49.0	40,040	1.014	56.1	44.7	27.0	40.9	173.50	11.320	0.084	125.00	4.94
2000-8	19-Sep-02	84	Balanced	215	450	591	1,298	3.28	57.4	0.65	2738	38.4	23.4	49.0	40,040	1.012	57.1	44.3	27.1	44.3	173.10	11.300	0.084	124.00	4.93
2000-8	21-Sep-02	86	Balanced	215	450	591	1,182	4.37	90.8	0.61	2568	37.9	21.0	48.5	40,040	1.038	59.1	54.0	26.6	43.5	163.10	10.650	0.08	122.00	3.90
2000-8	22-Sep-02	87	Balanced	215	450	591	1,208	4.25	84.4	0.62	2607	37.9	21.7	49.0	40,040	1.027	59.1	52.7	26.6	42.9	165.10	10.780	0.08	122.00	4.03
2000-8	23-Sep-02	88	Balanced	215	450	591	1,264	3.90	67.1	0.64	2695	38.8	22.9	48.0	40,040	1.025	59.1	49.7	26.8	41.4	171.10	11.150	0.085	127.00	4.36
2000-8	24-Sep-02	89	Balanced	215	450	591	1,243	3.94	70.2	0.63	2664	38.5	22.8	48.5	40,040	1.020	59.1	50.1	26.8	41.8	169.70	11.070	0.083	125.00	4.25
2000-8	25-Sep-02	90	Balanced	215	450	588	1,334	3.47	58.5	0.66	2788	39.4	24.7	48.0	40,040	0.976	59.1	44.5	26.3	41.3	170.90	11.140	0.085	125.00	4.88
2000-8	26-Sep-02	91	Balanced	215	450	602	1,312	3.24	73.9	0.65	2759	38.9	24.3	48.5	40,040	0.956	59.1	42.7	26.4	42.4	170.30	11.110	0.084	121.00	4.92
2000-8	27-Sep-02	92	Balanced	215	450	602	1,303	3.24	76.8	0.65	2737	39.5	24.4	47.5	40,040	0.965	59.1	43.0	26.5	42.4	170.30	11.110	0.085	124.00	4.90
2000-8	29-Sep-02	94	Balanced	215	450	678	1,190	3.70	129.3	0.64	2692	37.8	23.1	50.0	40,040	1.031	59.1	48.9	27.4	45.7	177.80	11.630	0.085	124.00	4.30

overall catalyst deactivation rate during the three months of operation since the completion of the second in-situ activation was about 0.2% to 0.3% per day. This is a significantly lower deactivation rate than the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day), and may be related to the selection of operating conditions, the performance of the adsorbent in the 29C-40 catalyst guard bed, and the removal of carbon steel components from the reactor during an inspection that was performed in June of 2002. As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. (This run was performed on CO-rich syngas derived from natural gas at a reactor temperature of 250°C.)

Due to this low catalyst deactivation rate, the practice of temperature programming was not required during the reporting period (temperature programming involves the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor volumetric productivity).

Samples of spent and recently activated fresh catalyst were collected during the quarter to determine changes in levels of trace contaminants (such as iron, nickel, sulfur, and arsenic) that are known poisons to methanol synthesis catalysts. These samples will be analyzed and reported when available.

29C-40 Catalyst Guard Bed Performance

As noted in Technical Progress Report No. 32, a fresh quantity of adsorbent (activated carbon impregnated with copper oxide) was charged to the 29C-40 catalyst guard bed in June of 2002. This material was chemically reduced using dilute syngas in nitrogen. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or H_2 to copper metal and either CO_2 or H_2O). The catalyst guard bed was placed into service on 27 June 2002.

During the period of 27 June 2002 to 03 July 2002, gas sampling was performed to assess the initial performance of the adsorbent. The analytical techniques involved analyzing the guard bed outlet stream for arsine using standard techniques. The outlet concentration of arsine was determined to be less than the detection limit of the analysis (2 ppbv). These results provide evidence that the catalyst guard bed was performing well during the first week of operation.

Based upon results over the past year, an operating schedule for the adsorbent was developed. Fresh adsorbent can be used for about two months before breakthrough of arsine can be expected. A thermal treatment can then be performed on the adsorbent to provide increased capacity for arsine removal, and the material can be used for another month before the adsorbent needs to be replaced. Based upon this timetable, a thermal treatment was completed on 29 August 2002. The catalyst guard bed was brought back onstream on 30 August 2002. It is planned to replace the used material with a fresh charge in early October of 2002.

Sparger Resistance

The performance of the gas sparger, which was designed by Air Products and first installed into the LPMEOHTM Reactor prior to the restart of the LPMEOHTM Demonstration Unit in March of 1999, was monitored. The device had been inspected and cleaned during the June 2002 outage prior to the second in-situ activation of methanol synthesis catalyst. Appendix C, Figure 2 plots the average daily sparger resistance coefficient for the fourth catalyst campaign. The data for this plot, along with the corresponding average pressure drop, are also included in Table D.3-1. The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device. The performance of the sparger will continue to be monitored closely for any changes.

D.4 Planning and Administration

A revision to a draft report that provides publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport was sent to DOE for comment. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 September 2002, are included in Appendix D. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. One hundred percent (100%) of the \$40 million of funds forecast for the Kingsport portion of the LPMEOHTM Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 2002. Ninety-eight percent (98%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2002.

The monthly reports for July, August, and September were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

E. Planned Activities for the Next Quarter

- Continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Focus activities on temperature programming to maintain the required methanol productivity, monitoring catalyst activity, assessing the performance of the catalyst guard bed, and monitoring the performance of the gas sparger in the reactor.
- Schedule a Project Review Meeting with DOE.

F. Conclusion

The LPMEOHTM Demonstration Unit operated at 100% availability during this quarter. There were two syngas interruptions that were experienced on 18 August 2002 (16.1 hours duration) and 19 August 2002 (12.4 hours duration).

During this quarter, the LPMEOHTM Demonstration Unit was operated at a reactor temperature of 214-215°C and a pressure of 450 psig. The flowrate of Balanced Gas was controlled at an average value of 551 KSCFH during this time. These conditions have been used since the start of the current catalyst campaign and were selected as a result of the excellent initial activity of the methanol synthesis catalyst following the in-situ activation in June of 2002.

A catalyst deactivation rate of 0.52% per day was calculated for the period 28 June 2002 to 02 August 2002 (35 days). These results are excellent since this deactivation rate includes the period of hyperactivity that is normally encountered with freshly activated methanol synthesis catalysts.

Beginning on 05 August 2002, Balanced Gas composition began to vary greatly for the remainder of the reporting period. This compositional variation resulted in periods of operation with both CO-rich and H₂-rich syngas. Due to these variations in concentration and their impact on the accuracy of the kinetic model, a catalyst deactivation rate could not be calculated during August and September of 2002. However, it was estimated that the overall catalyst deactivation rate during the three months of operation since the completion of the second in-situ activation was about 0.2% to 0.3% per day. This is a significantly lower deactivation rate than the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day), and may be related to the selection of operating conditions, the performance of the adsorbent in the 29C-40 catalyst guard bed, and the removal of carbon steel components from the reactor during an inspection that was performed in June of 2002. As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. (This run was performed on CO-rich syngas derived from natural gas at a reactor temperature of 250°C.)

Due to this low catalyst deactivation rate, the practice of temperature programming was not required during the reporting period (temperature programming involves the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor volumetric productivity).

Samples of spent and recently activated fresh catalyst were collected during the quarter to determine changes in levels of trace contaminants (such as iron, nickel, sulfur, and arsenic) that are known poisons to methanol synthesis catalysts. These samples will be analyzed and reported when available.

As noted in Technical Progress Report No. 32, a fresh quantity of adsorbent (activated carbon impregnated with copper oxide) was charged to the 29C-40 catalyst guard bed in

June of 2002. This material was chemically reduced using dilute syngas in nitrogen. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or H_2 to copper metal and either CO_2 or H_2O). The catalyst guard bed was placed into service on 27 June 2002.

During the period of 27 June 2002 to 03 July 2002, gas sampling was performed to assess the initial performance of the adsorbent. The analytical techniques involved analyzing the guard bed outlet stream for arsine using standard techniques. The outlet concentration of arsine was determined to be less than the detection limit of the analysis (2 ppbv). These results provide evidence that the catalyst guard bed was performing well during the first week of operation.

Based upon results over the past year, an operating schedule for the adsorbent was developed. Fresh adsorbent can be used for about two months before breakthrough of arsine can be expected. A thermal treatment can then be performed on the adsorbent to provide increased capacity for arsine removal, and the material can be used for another month before the adsorbent needs to be replaced. Based upon this timetable, a thermal treatment was completed on 29 August 2002. The catalyst guard bed was brought back onstream on 30 August 2002. It is planned to replace the used material with a fresh charge in early October of 2002.

The performance of the gas sparger, which was designed by Air Products and first installed into the LPMEOHTM Reactor prior to the restart of the LPMEOHTM Demonstration Unit in March of 1999, was monitored. The device had been inspected and cleaned during the June 2002 outage prior to the second in-situ activation of methanol synthesis catalyst. The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device. The performance of the sparger will continue to be monitored closely for any changes.

During the reporting period, a total of 4,572,193 gallons of methanol was produced at the LPMEOHTM Demonstration Unit. Since startup, about 99.8 million gallons of methanol have been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

A revision to a draft report that provides publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport was sent to DOE for comment. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems.

One hundred percent (100%) of the \$40 million of funds forecast for the Kingsport portion of the LPMEOHTM Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 2002. Ninety-eight percent (98%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2002.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

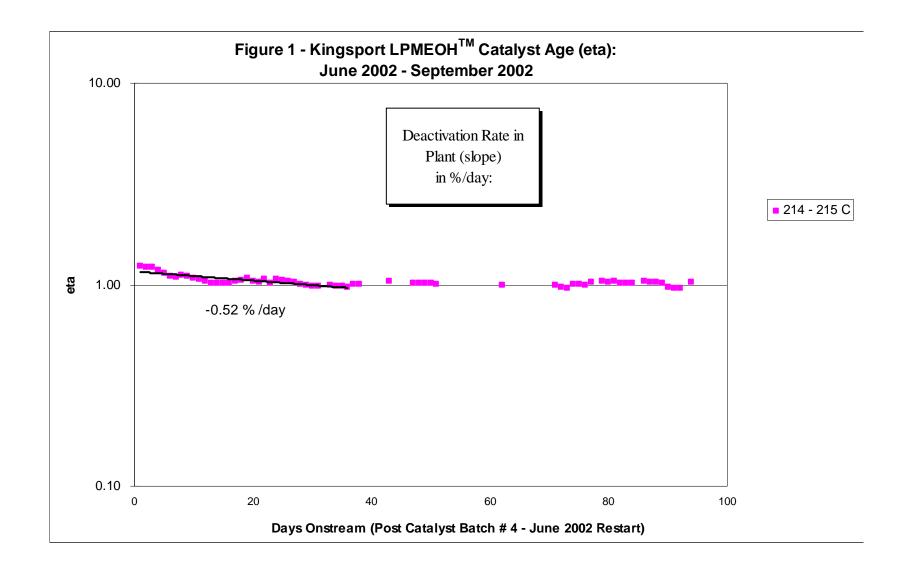
APPENDIX B - SAN	MPLES OF DETAIL	LED MATERIAL BA	ALANCE REPORTS

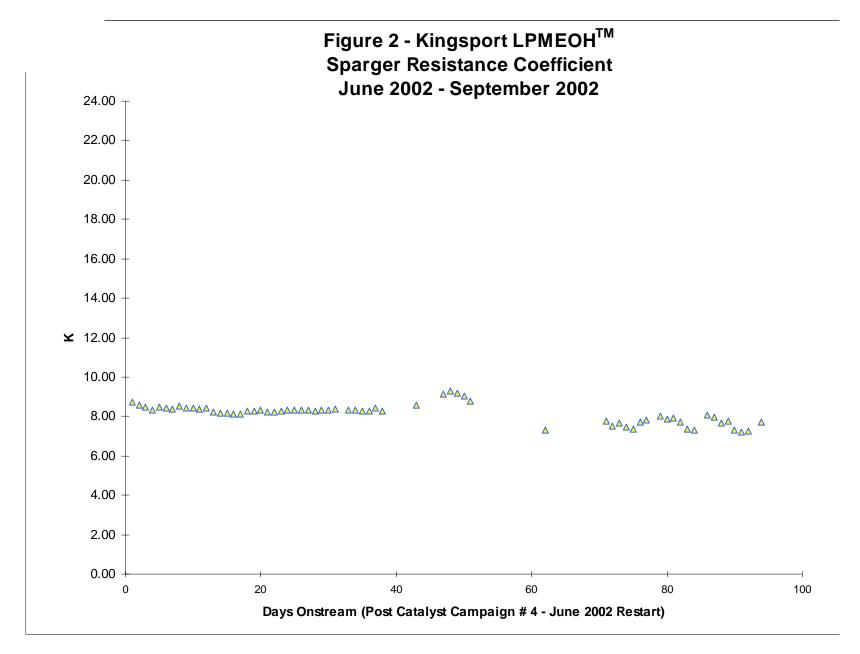
APPENDIX C - RESULTS OF DEMONSTRATION UNIT OPERATION

- Table 1 Summary of LPMEOH $^{\rm TM}$ Demonstration Unit Outages July/September 2002
- Figure 1 Catalyst Age (η): June 2002 September 2002
- Figure 2 Sparger Resistance Coefficient vs. Days Onstream (June 2002 September 2002)

 ${\bf Table~1} \\ {\bf Summary~of~LPMEOH^{\rm TM}~Demonstration~Unit~Outages~-~July/September~2002}$

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
7/1/02 00:00 8/19/02 04:50 8/20/02 00:55	8/18/02 12:44 8/19/02 12:33 9/30/02 23:59	1164.7 7.7 1007.1	16.1 12.4	Syngas Outage Syngas Outage End of Reporting Period
5	Total Operating Hours Syngas Available Hours Plant Availability, %		2179.5 2179.5 100.00	





APPENDIX D - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS